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SOLID STATE FORMATION OF ND:Y SUB 3 AL SUB 5 O SUB 12 (ND:YAG).(U)  
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# SOLID STATE FORMATION OF Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG)

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6 Solid State Formation of  
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DENNIS J. VIECHNICKI and JAROSLAV L. CASLAVSKY  
CERAMICS RESEARCH DIVISION

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ABSTRACT

The formation of 1.1 at. % Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) from mixed oxide starting powders after heating at 1650 C for 3 hours in air is reported for the first time. Cylindrical billets are produced with fired densities between 65% and 75% of theoretical suitable for use as meltstock for crystal growth by the heat exchanger method (HEM). Conditions for milling of the starting oxide powders to eliminate agglomerates which lead to formation of more volatile Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub> and YAlO<sub>3</sub> are discussed. A comparison is made between mixed oxides and hydrated salts for use as starting materials.

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## INTRODUCTION

The growth of large sapphire single crystals by the heat exchanger method (HEM)<sup>1</sup> is facilitated by the availability of high purity sapphire crackle, i.e., cracked vernueil grown sapphire boules with impurity levels of less than 40 ppm, that can be packed into a crucible at an efficiency of 70%. This packing efficiency is needed to maximize crystal height without having a "boil over" because of the 22% volume expansion of sapphire upon melting.<sup>2</sup> During a program involving growth of large (1 to 2 kg) single crystals of 1.1 at. % Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) by HEM, no Nd:YAG crackle was commercially available in large quantities. It was decided to make the meltstock in-house by sintering isostatically pressed cylindrical billets of mixed oxide powders to 65% to 75% of theoretical density. After firing, the billets would be just small enough to slip inside the crucible.

Additional requirements for the meltstock were that it be moisture-free, and that it be completely reacted to contain only the one compound, Nd:YAG. Crystal growth by HEM is done in vacuo or inert atmosphere in a graphite resistance furnace. It was found that to grow high quality spinel<sup>3</sup> with minimum crucible-melt reaction and with minimum weight loss during growth, it was necessary to react the MgO and Al<sub>2</sub>O<sub>3</sub> completely to form MgAl<sub>2</sub>O<sub>4</sub> prior to melting.

A concurrent investigation had showed that the presence of unreacted Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>, and the other binary compounds, YAlO<sub>3</sub> and Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub>,<sup>4</sup> all with different high temperature volatilization rates, caused the melt to deviate from stoichiometry.<sup>5</sup> While Messier and Gazza<sup>6</sup> reported that prolonged heating of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> mixtures between 1500 C and 1600 C would not produce pure YAG free of the metastable compounds, they did not investigate the effects of milling on the formation of YAG. Reijnen<sup>7</sup> investigated the effects of milling on the formation of NiFe<sub>2</sub>O<sub>4</sub> from mixtures of NiO and Fe<sub>2</sub>O<sub>3</sub>. Some Fe<sub>3</sub>O<sub>4</sub> was formed when the NiO and Fe<sub>2</sub>O<sub>3</sub> did not react completely. The Fe<sub>3</sub>O<sub>4</sub> was finally eliminated by more milling of the starting mixtures. It was felt that similar effects might be found with mixtures of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>. Therefore the amount of milling needed to insure proper compound formation was investigated.

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6. MESSIER, D. R., and GAZZA, G. E. *Synthesis of MgAl<sub>2</sub>O<sub>4</sub> and Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> by Thermal Decomposition of Hydrated Nitrate Mixtures*. Am. Ceram. Soc. Bull., v. 51, no. 9, 1972, p. 692-694, 697; also Army Materials and Mechanics Research Center, AMMRC TR 73-2, January 1973.
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## EXPERIMENTAL

Starting materials were high purity oxides, 99.992% pure alumina,\* 99.999% pure yttria,<sup>†</sup> and 99.0% pure neodymia.<sup>‡</sup> These were weighed in correct proportions in 300-gram batches to form Nd:YAG, i.e., 37.1 mol %  $Y_2O_3$ /62.5 mol %  $Al_2O_3$ /0.4 mol %  $Nd_2O_3$ . The powders were either mixed by allowing ethanol to boil away in a 2-gallon ball mill made of high alumina, or milled in the same mill under several different conditions. The time of milling, the number of 1/2"-diameter high alumina balls, and the milling fluid, ethanol or tertiary butanol, were systematically varied. Conditions used are listed on Figures 1 and 2. After drying, the powders were placed in a rubber bag and isostatically pressed at 30,000 psi to form rods 15 mm in diameter by 100 mm long. These were cut into 15-mm lengths with a dry diamond saw, and their bulk densities were taken by weighing them and then measuring them with a micrometer. Volumes were calculated from the physical dimensions. The rods were heated at 1650 C in air in a zirconia felt-lined  $MoSi_2$  furnace for 1, 3, 10, or 10-1/4 hours. Weight loss, shrinkage, and bulk density determinations were made after firing. Phases present were determined by X-ray powder diffraction on crushed samples. The morphology of selected powders before and after milling were studied using a scanning electron microscope (SEM).

## RESULTS AND DISCUSSION

Weight losses after firing ranged from 1.64% to 1.94%, probably dependent upon how well the material was dried after milling. The effect of milling upon compound formation after firing at 1650 C in air for various times is seen in Figure 1.

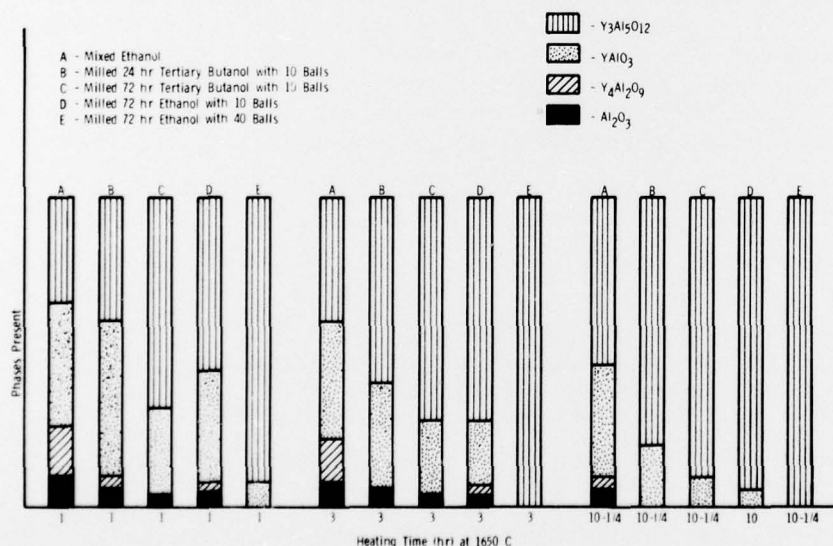


Figure 1. Phases present after heating powder mixtures of 37.1 mol %  $Y_2O_3$ /62.5 mol %  $Al_2O_3$ /0.4 mol %  $Nd_2O_3$ .

\*0.3  $\mu m$  Extra Pure, Adolf Meller Company, Providence, Rhode Island 02904

<sup>†</sup>563  $Y_2O_3$ , Molycorp, White Plains, New York 10604

<sup>‡</sup> $Nd_2O_3$  Code 629.9, American Potash and Chemical Corp., Rare Earths Division, West Chicago, Illinois 60185.

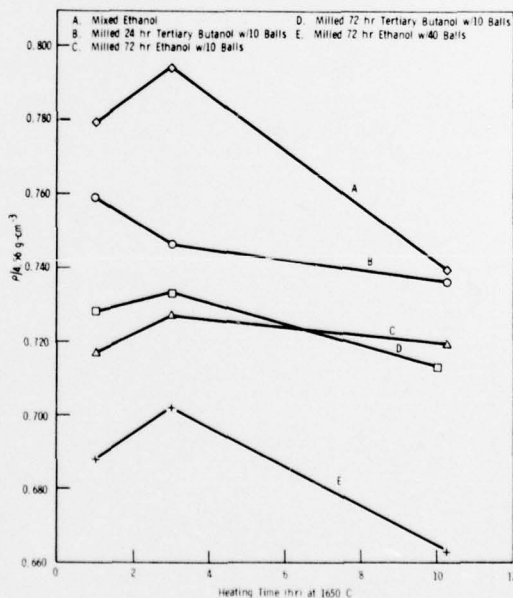


Nd:YAG can be formed by heating mixed oxides in air at 1650 C for 3 hours. Warshaw and Roy<sup>8</sup> reported forming YAG from hydroxides at 1700 C, but neither they nor Messier and Gazza<sup>6</sup> could form pure YAG from mixed oxides. The longer the powder is milled, the more balls used during milling, and the longer it is heated at 1650 C the more readily YAG is formed. There is a certain minimum milling, 72 hours with 40 balls for a 300-gram batch, that must be done to prevent formation of  $Y_4Al_2O_9$  and all but a trace of  $YAlO_3$ . Once  $Y_4Al_2O_9$  is formed it exists until 1940 C, the eutectic temperature between  $Y_4Al_2O_9$  and  $Y_2O_3$ .<sup>4</sup> This is 30° below the melting point of YAG, 1970 C,<sup>8</sup> a temperature that we have confirmed in this investigation. Some melting of the billet is observed at 1940 C if it contains  $Y_4Al_2O_9$ . A fully homogenized single compound billet of YAG melts uniformly at 1970 C. This premelting may be responsible for the range of melting points reported, i.e., Toropov et al. 1930 C,<sup>4</sup> Warshaw and Roy<sup>8</sup> and this investigation 1970 C.

$Y_4Al_2O_9$  is more volatile than YAG. Under similar conditions, where a YAG melt would be very still and where the differences between initial and final weights of the change would indicate a 0.5% weight loss, a  $Y_4Al_2O_9$  melt would bubble continuously and show a 10.7% weight loss. Thus any billet containing  $Y_4Al_2O_9$  results in a Nd:YAG boule that is off stoichiometry and contains second-phase scattering centers. This was confirmed optically and by X-ray powder diffraction analysis.<sup>5</sup> A similar argument, but with less drastic results, may be made for having  $YAlO_3$  in the starting material. Formation of YAG was not enhanced when using tertiary butanol as a milling fluid. Ethanol was preferred because it was easier to work with.

Figure 2 shows the results of the densification as a function of milling and calcining times. The fired density divided by 4.56 g/cm<sup>3</sup>, the theoretical density of  $Y_3Al_5O_{12}$ ,<sup>9</sup> is shown as a function of calcining time at 1650 C. The curves show a rapid densification followed by a decline in density. This can be correlated with

Figure 2. Densification of sintered billets of YAG expressed as bulk density of Nd:YAG/theoretical density of YAG as a function of heating time in air at 1650 C.



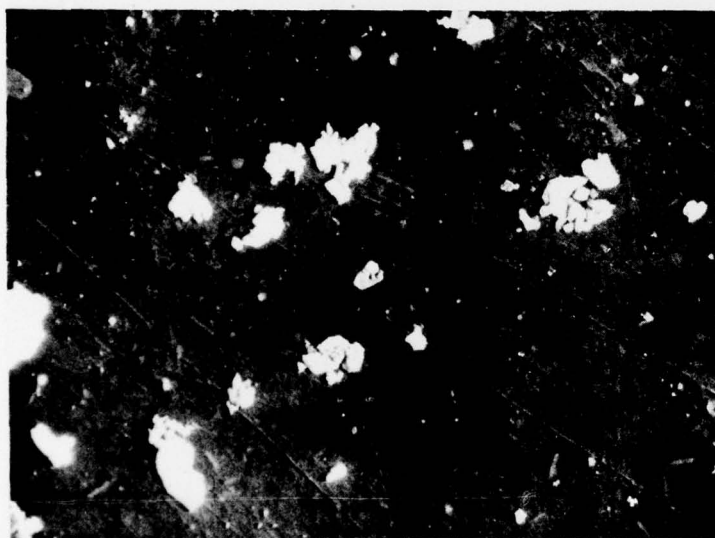
8. WARSHAW, I., and ROY, R. *Stable and Metastable Equilibria in the Systems  $Y_2O_3 \cdot Al_2O_3$  and  $Gd_2O_3 \cdot Fe_2O_3$* . J. Am. Ceram. Soc., v. 42, no. 9, 1959, p. 434-438.

9. KLEIN, P. H., and CROFT, W. J. *Thermal Conductivity, Diffusivity, and Expansion of  $Y_2O_3$ ,  $Y_3Al_5O_{12}$ , and  $LaF_3$  in the Range 77°-300° K.* J. Appl. Phys., v. 38, no. 4, 1967, p. 1605.

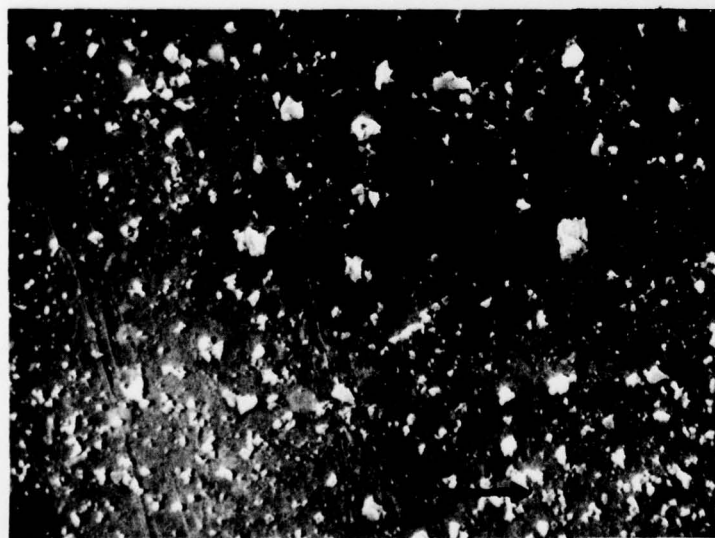


the phases present shown in Figure 1. The initial compounds to form are  $YAlO_3$ ,  $\rho = 5.36 \text{ g/cm}^3$ ,<sup>10</sup> which is the densest compound in the binary, and  $Y_4Al_2O_9$ ,  $\rho = 4.41 \text{ g/cm}^3$ .<sup>4</sup> This results in rapid apparent densification. As more YAG is formed and the amount of  $YAlO_3$  decreases, the measured density decreases. More milling retards the initial metastable formation of  $YAlO_3$  and  $Y_4Al_2O_9$  and results in lower overall densities. Thus the more YAG the material contains, the lower its density.

This argument is supported when the dispersed powders are examined with an SEM before and after milling. An unmilled stoichiometric mixture of powders is seen in Figure 3a. Large agglomerates of mainly  $Y_2O_3$  and  $Al_2O_3$  powders are evident. These are broken up after milling, resulting in a more uniform particle size (Figure 3b).



a. Mixed in Ethanol



b. Milled 72 Hours in Ethanol

Figure 3. Scanning electron microscope photos of starting oxide powder mixtures for growth of Nd:YAG. Mag. 1000X  
19-066-288/AMC-77

10. DIEHL, R., and BRANDT, G. *Crystal Structure Refinement of  $YAlO_3$ , a Promising Laser Material*. Mat. Res. Bull., v. 10, 1975, p. 85-90.

These large agglomerates can react together to form unwanted  $Y_4Al_2O_9$  and  $YAlO_3$ , depending upon localized concentrations of  $Al_2O_3$  and  $Y_2O_3$ .

While rapid compound formation is possible when hydrated salts such as  $Al(NO_3)_3 \cdot 9H_2O$  and  $Y(NO_3)_3 \cdot 6H_2O$  are used as precursors and then decomposed,<sup>6,8</sup> there are drawbacks as deduced from Table 1. Much larger quantities of salts are needed to produce equivalent weight crystals. Since the cost per kilogram is roughly the same as for the oxide powders, the use of salts is more expensive. Waste product disposal may be a concern in a production operation. The oxide powders are cleaner. Also there are fewer residual anion impurities left in the sintered billet when oxide powders are the starting material.\* This results in a higher quality crystal.

Table 1. COMPARISON OF MAKING YAG SINGLE CRYSTALS FROM VARIOUS STARTING MATERIALS

<u>Factors</u>	<u>Decomposition of <math>Al(NO_3)_3 \cdot 9H_2O</math> and <math>Y(NO_3)_3 \cdot 6H_2O</math></u>	<u>Mixed Oxide Powders</u>
Compound formation	Rapid	Slow
<u>Weight of starting material</u> Weight of crystal	7.36	1.02
<u>Volume of starting material</u> Volume of crystal	33	19
Cost	7 to 8 times more expensive Cost/kg salt = Cost/kg oxide powder but 7.36 times more salt is required to produce the same weight crystal.	
Waste products	$NO_x$ , residual anion impurities in final product	Ethanol vapors

#### ACKNOWLEDGMENTS

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\*Private communications with D. R. Messier and G. E. Gazza, 25 August 1977.

<p>Army Materials and Mechanics Research Center Watertown, Massachusetts 02172 SOLID STATE FORMATION OF Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) — Dennis J. Viechnicki and Jaroslav L. Caslavsky</p> <p>Technical Report AMMRC TR 78-7, February 1978, 7 pp — illus-table, D/A Project 1T162105AH84, AMCMS Code 612105</p> <p>The formation of 1.1 at. % Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) from mixed oxide starting powders after heating at 1650 C for 3 hours in air is reported for the first time. Cylindrical billets are produced with fired densities between 65% and 75% of theoretical suitable for use as meltstock for crystal growth by the heat exchanger method (HEM). Conditions for milling of the starting oxide powders to eliminate agglomerates which lead to formation of more volatile Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub> and YAlO<sub>3</sub> are discussed. A comparison is made between mixed oxides and hydrated salts for use as starting materials.</p>	<p>AD — UNCLASSIFIED UNLIMITED DISTRIBUTION</p> <p>Key Words Yttrium-aluminum-garnet formation Compound formation Densification</p>	<p>Army Materials and Mechanics Research Center Watertown, Massachusetts 02172 SOLID STATE FORMATION OF Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) — Dennis J. Viechnicki and Jaroslav L. Caslavsky</p> <p>Technical Report AMMRC TR 78-7, February 1978, 7 pp — illus-table, D/A Project 1T162105AH84, AMCMS Code 612105</p> <p>The formation of 1.1 at. % Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) from mixed oxide starting powders after heating at 1650 C for 3 hours in air is reported for the first time. Cylindrical billets are produced with fired densities between 65% and 75% of theoretical suitable for use as meltstock for crystal growth by the heat exchanger method (HEM). Conditions for milling of the starting oxide powders to eliminate agglomerates which lead to formation of more volatile Y<sub>4</sub>Al<sub>2</sub>O<sub>9</sub> and YAlO<sub>3</sub> are discussed. A comparison is made between mixed oxides and hydrated salts for use as starting materials.</p>	<p>AD — UNCLASSIFIED UNLIMITED DISTRIBUTION</p> <p>Key Words Yttrium-aluminum-garnet formation Compound formation Densification</p>
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 Compound formation  
 Denification

The formation of 1.1 at. % Nd:Y<sub>3</sub>Al<sub>5</sub>O<sub>12</sub> (Nd:YAG) from mixed oxide starting powders after heating at 1650 C for 3 hours in air is reported for the first time. Cylindrical billets are produced with fired densities between 65% and 75% of theoretical suitable for use as meltstock for crystal growth by the heat exchanger method (HEM). Conditions for milling of the starting oxide powders to eliminate agglomerates which lead to formation of more volatile Y<sub>6</sub>Al<sub>2</sub>O<sub>9</sub> and YAlO<sub>3</sub> are discussed. A comparison is made between mixed oxides and hydrated salts for use as starting materials.